

# PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

### Fertilizers

We, Fisons Fertilizers Limited, a British Company, of Harvest House, Felixstowe, Suffolk, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to granular ammonium nitrate or ammonium sulphate nitrate, the caking tendency of which has been substantially reduced.

It is a well-known fact that fertilizer materials which contain nitrate ions are very subject to caking. Various remedial treatments have been proposed which involve coating the nitrate-containing fertilizer material with one of a number of powdered inert materials or with hydrophobic oils. Most surprisingly it has now been found that the caking tendency of granular nitrate-containing fertilizers can be considerably reduced if a small proportion of aluminium hydroxide or a basic aluminium salt is uniformly incorporated into the nitrate-containing fertilizer material.

Accordingly the present invention comprises a granular ammonium nitrate, or ammonium sulphate nitrate, containing 0.1 to 5.0% by weight based on the total weight of the composition of aluminium hydroxide or a basic aluminium salt uniformly distributed within the granules.

The proportion of aluminium hydroxide or basic aluminium salt in the fertilizer material is preferably equivalent to 0.15 to 0.35% aluminium in the final product.

The granular ammonium nitrate or ammonium sulphate nitrate fertilizers of the present invention may be prepared by a process which comprises feeding to a rotary granulator a solid ammonium salt selected from ammonium sulphate and ammonium nitrate, a water soluble aluminium salt, an aqueous solution of ammonium nitrate containing less than 20%

by weight of water and at an elevated temperature, recycle material as hereinafter defined and anhydrous ammonia, granulating the mixture at a temperature above 50°C and thereafter drying the granules so formed the amount of ammonia being such that the pH of a 10% solution by weight of the granules in water is in the range 4.5 to 5.0, and the amount of said aluminium salt, added being such as to provide 0.1% to 5.0% of aluminium hydroxide or basic aluminium salt in the final product.

The ammonia is added to reduce the acidity of the nitrate-containing fertilizer and convert the aluminium salt to the hydroxide or a basic salt. The ammonia is preferably added in such an amount that the pH of a 10% solution of the ammonium-nitrate or ammonium sulphate nitrate containing fertilizer in water is in the range 4.6—4.7.

The aluminium salt may be added to the granulator either as a powder or as a solution in water or dissolved in the ammonium nitrate solution. The aluminium salt is preferably aluminium sulphate or aluminium nitrate.

In order to reduce the caking tendency of the product as much as possible it is preferred to dry the granules to a water content of 0.1 to 5% preferably to a water content in the range 0.1 to 0.2% by weight. The drying of the granules is preferably carried out in a rotary drier through which a co-current or countercurrent stream of hot air is passed.

In a preferred embodiment of the present invention a process is provided for making non-caking ammonium sulphate nitrate in granular form which comprises feeding to a rotary granulator ammonium sulphate crystals, a water soluble aluminium sulphate, an aqueous solution of ammonium nitrate, containing less than 20% by weight of water and

at an elevated temperature, recycle material as hereinafter defined and anhydrous ammonia, granulating the mixture at a temperature above 50°C and, thereafter, drying the granules so formed, the amount of ammonia being such that the pH of a 10% solution by weight of the granules in water is in the range 4.5 to 5.0 and the amount of said aluminium salt added being such as to provide 0.1% to 5.0% of aluminium hydroxide or basic aluminium salt in the final product. Preferably the ammonia is introduced under the bed of material being granulated. Recycle material comprises fines, that is granules which are less than the product size of from 1.0 mms. to 3.5 mms, and ground oversize.

The following examples in which parts are by weight are given to illustrate the process of the present invention.

#### EXAMPLE 1

An ammonium sulphate nitrate fertilizer containing 20% by weight of nitrogen was manufactured continuously by the addition to a rotary granulator of 61.3 parts per hour of ammonium sulphate crystals (70% passing 18 B.S.S. and 24% passing 30 B.S.S.); 40.5 parts per hour of 91% ammonium nitrate solution at 110°C; 2.2 parts per hour of hydrated aluminium sulphate; 0.25 parts per hour of anhydrous ammonia, together with 80 parts per hour of recycle of 1.0 mm mean size. The temperature of the mixture in the rotary granulator was 60—65°C and the mean free water content was 2.5—3.5%.

The granules were dried co-currently in a rotary dryer using heated air at 325—335°C yielding a granule dryer exit temperature of 110—120°C; 60% of the granules were in the size range 3.5—1.75 mms with a free moisture content of 0.15%.

After subsequent cooling and screening the granular product was examined for caking propensity using the test procedure described in J.S. Food and Ag. 1957, 10. 577—591. After 7 days storage at 6 p.s.i. pressure and 35°C an acceptable level of caking of 12 p.s.i. was determined.

A similar product prepared without the use of an aluminium salt and ammonia was tested under identical conditions and a completely unacceptable caking index value in excess of 80 p.s.i. was measured.

#### EXAMPLE 2

An ammonium sulphate nitrate fertilizer containing 30% by weight of nitrogen was produced continuously by the addition to a rotary granulator of 30 parts per hour of ammonium sulphate crystals (96% passing 18 B.S.S. and 4% passing 30 B.S.S.); 73 parts per hour of 92% ammonium nitrate solution at 120°C in which there were dissolved 2.85 parts per hour of hydrated aluminium sulphate; 0.25 parts per hour of anhydrous ammo-

nia together with 160 parts per hour of dried recycle of mean size 0.8 mm.

The temperature of the granules in the rotary granulator was 70—75°C and their mean free water content was 1.8—2.5%.

The granules were dried using heated air at 300—320°C giving a granule dryer exit temperature of 105—110°C. The granules were 50% in the range 3.5—1.75 mms. and contained 0.12% free moisture content.

After cooling and screening the product was examined for caking propensity as in Example 1 and an acceptable index of 8 p.s.i. was measured. As a comparative example an ammonium sulphate nitrate product of similar moisture and nitrogen content without aluminium salt and ammonia addition was tested for caking propensity; the unacceptable index value of 95 p.s.i. was determined.

#### WHAT WE CLAIM IS:—

1. A granular ammonium nitrate or ammonium sulphate nitrate, containing 0.1% to 5.0% by weight based on the total weight of the composition of aluminium hydroxide or a basic aluminium salt uniformly distributed within the granules.

2. A granular ammonium nitrate or ammonium sulphate nitrate as claimed in Claim 1 which contains 0.15% to 0.35% by weight based on the total weight of the composition of aluminium hydroxide or basic aluminium salt.

3. A process for preparing granular ammonium nitrate or ammonium sulphate nitrate as claimed in claim 1 which comprises feeding to a rotary granulator a solid ammonium salt selected from ammonium sulphate and ammonium nitrate, a water-soluble aluminium salt, an aqueous solution of ammonium nitrate containing less than 20% by weight of water and at an elevated temperature, recycle material as hereinbefore defined and anhydrous ammonia, granulating the mixture at a temperature above 50°C and, thereafter, drying the granules so formed, the amount of ammonia being such that the pH of a 10% solution by weight of the granules in water is in the range 4.5 to 5.0 and the amount of said aluminium salt added being such as to provide 0.1% to 5.0% of aluminium hydroxide or basic aluminium salt in the final compound.

4. A process as claimed in Claim 3 wherein the aluminium salt is fed to the rotary granulator dissolved in the ammonium nitrate solution.

5. A process as claimed in Claim 3 wherein the aluminium salt is fed as a solution in water to the rotary granulator.

6. A process as claimed in Claim 3 wherein the aluminium salt is fed as a powder to the rotary granulator.

7. A process as claimed in any of claims

- 3 to 6 wherein the aluminium salt is aluminium sulphate or aluminium nitrate.
8. A process as claimed in any of claims 3 to 7 wherein the granules are dried to a water content of 0.1 to 5% by weight.
9. A process as claimed in any of claims 3 to 7 wherein the granules are dried to a water content in the range 0.1 to 0.2% by weight.
10. A process for preparing non-caking ammonium sulphate nitrate in granular form as claimed in claim 1 which comprises feeding to a rotary granulator ammonium sulphate crystals, aluminium sulphate, an aqueous solution of ammonium nitrate containing less than 20% by weight of water and at an elevated temperature, recycle material as hereinbefore defined and anhydrous ammonia, granulating the mixture at a temperature above 50°C and thereafter drying the granules so formed, the amount of ammonia being such that the pH of a 10% solution by weight of the granules in water is in the range 4.5 to 5.0 and the amount of aluminium sulphate added being such as to provide 0.1% to 5.0% of aluminium hydroxide or basic aluminium salt in the final product.
11. A process for preparing a granular ammonium nitrate or ammonium sulphate nitrate containing 0.1% to 5.0% by weight of aluminium hydroxide or basic aluminium salt based on the total weight of the composition as claimed in claim 3 substantially as hereinbefore described.
12. A granular ammonium nitrate or ammonium sulphate nitrate containing 0.1% to 5.0% by weight based on the total weight of the composition of aluminium hydroxide or basic aluminium salt when prepared by the process as claimed in any of claims 3 to 11.
13. A granular ammonium nitrate or ammonium sulphate nitrate as claimed in claim 1 substantially as hereinbefore described.

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